# A Specimen Preparation Technique for the Crystal Habit

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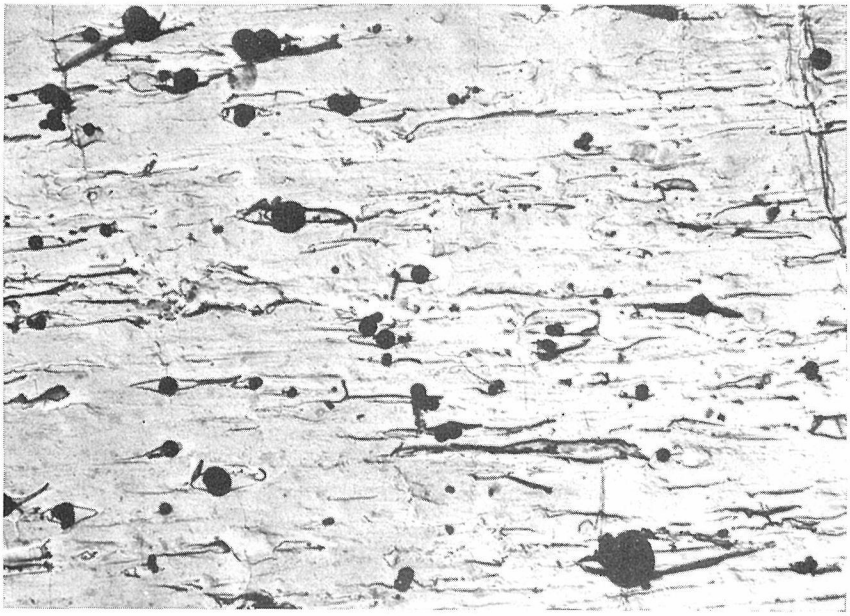
In the electron micrographs of each samples at the time of elongation, many parallel streaks running to the tensile direction were observed, whereas they could not be observed in the unelongated original rubber. Cleavages were also observed clearly at both edges of filler particles at the tensile direction as shown in Fig. 1. At the time of elongation, it is conceivably possible that there grow cleavages at both edges of filler particles. The existense of the cleavages, which has already been speculated by Schippel (1920), was proved at the surface of rubber, and its shape was demonstrated.



Electron micrograph of the elongated rubber loaded with carbon black. Cleavages at both edges of filler particles are observed. Metheyl methacryl-SiO two-step casting replica was used. (×16,000)

## A Specimen Preparation Technique for the Crystal Habit Analysis of Fibrous Crystals

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A simple technique is presented for the preparation of a specimen containing "rafts" of slender crystals to study the fiber period, growth direction, crystal habit, etc.

A small amount of specimen powder is ultrasonically dispersed in an organic medium which is not soluble in water and does not act as a solvent for the powder. A small quantity of suspension is floated on a surface of water. It spreads all over the surface and the needle-like crystals flow along local streaming

lines of the suspension, forming "rafts" of the crystals. The crystals are transferred onto a surface of a grid covered with Formvar film and dried in a desiccator.

When the crystal is an organic compound which can not be wetted by water, the water soluble solvent, such as pyridine or acetone, can be used as the suspension medium. The special hydrosol, such as vanadium pentoxide sol, can also be kept afloat on a surface of water to make "rafts" of crystals.

Electron microdiffraction of a part of the raft mentioned above shows parallelly layered fiber pattern. For the analysis of the pattern, the rotation effect of the image must be taken into account. The maximum period of the lattice structure along the fiber axis $y_0$ is given by the following equation,

$$y_0 = \lambda L_f / h_0$$

where $L_f$ is the final camera length, $\lambda$ is the wave length of the electron beam, and $h_0$ is the distance between the two neighbouring layer lines of the fibre pattern. The constant value of $\lambda L_f$ was obtained with mico-single crystals of gold used as a reference. From the fiber pattern the crystal habit and the orientation of the lattice in the crystal can be identified. When the lattice structure is not known, the fiber diagram analysis presents some powerful clue to find the element of cell constants.

## Examples

The crystal in aged Biltz sol, a kind of vanadium pentoxide sol, is fibrous. When a drop of the sol was set afloat on a surface of water, a tactoid was formed. An electron diffraction pattern obtained from the tactoid was a fiber diagram, in which (020) and its higher order reflections lay on the meridian. The parallel layer lines suggest that the fiber axis is b-axis. The maximum lattice period was calculated as 3.56 Å and was in good agreement with the value of $b_0$ for vanadium pentoxide. The fiber axis of the fibrous crystal in Biltz sol was decided as b-axis.

In the case of $\beta$-copper-phthalocyanine, $y_0$ was 4.76 Å and was in good agreement with the cell dimension of b-axis, 4.79 Å, which was determined by Robertson. This shows that the direction of the longest edge of the lath-shape crystal is the b-axis. The spots of $(20\bar{1})$ and its higher order reflections could be often found in a diffraction pattern fairly clearly. This indicates that the habit of the lathy micro-crystals is also the same as the macro-crystals reported by Robertson. Though many crystals had somewhat irregular perimeters, the ends of the long flakes were always perpendicular to the axis. This plane corresponds to (010) and the large habit surface corresponds to (001).

Zinc-phthalocyanine was also slender crystal and the lattice period along the direction of the extension was found to be 5.41 Å. Owing to the wide habit surface of the ribbon-like crystal, the single crystal pattern was often superimposed especially on the equator. This shows that the lattice plane, which is almost perpendicular to the largest crystal habit surface on which the crystal lies, runs parallelly along the direction of the longest edge of the ribbon at an interplanar spacing of 12.6 Å. This lattice plane presents a large advantage for the direct

observation of the crystal lattice by an electron microscope, owing to the wideness of the spacing and the inclination to the incident electron beam.

## Comparison of the Particle Sizes of Powders Measured by Various Methods

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Determination of the particle size of powder is very important for industrial and theoretical use, and, therefore, various measuring methods has been developed. The particle sizes of various powders measured by sedimentation, adsorption and air-permeability methods were compared with the results by the electron microscopy. The powders used in this study were carbon black, calcium carbonate, titanium dioxide, rouge, and clay.

The mean particle size measured by the absorption or air-permeability method is average surface diameter $d_3$, depending on the specific surface area of powders. To compare with the particle sizes by these method, the value of $d_3$, i. e. $\Sigma nd^3/\Sigma nd^2$, must accordingly be used after conversion through the size distribution curve by the electron microscope. In the case of the powders having especially uniform shape and size, for example, calcium cabonate prepared by the chemical reaction all of the values of mean particle sizes by various methods were almost the same. For the ordinary powders, however, the values measured by various methods did not agree, as shown in Table 1. This was explained, from the observation of the particle shape with electron microscope, to be due to other properties of powder.

Table 1. Particle size measured by various methods ($\mu$).

| Sample | Electron-microsocpe | Sedimen-tation | Permea-bility | Adsorption | |
|---|---|---|---|---|---|
| | | | | Liquid | B E T |
| Carbon black F | 0.12 | —— | 0.26 | —— | 0.13 |
| Carbon black C | 0.024 | —— | —— | —— | 0.015 |
| $CaCO_3$-A | 3.4 | 3.5 | 3.5 | 3.4 | —— |
| $CaCO_3$-B | 1.6 | 1.8 | 1.7 | 1.58 | —— |
| $CaCO_3$-U | 0.062 | —— | —— | 0.869 | —— |
| $TiO_2$-Z | 0.19 | 0.52 | —— | 0.40 | —— |
| $TiO_2$-D | 0.23 | 0.69 | —— | 0.82 | —— |
| Rouge-K | 0.15 | —— | —— | 0.21 | —— |
| Rouge-T | 1.29 | 0.85 | —— | 0.62 | —— |
| Rouge-N | 14.88<br>0.52 | 10.2 | —— | 0.69 | —— |
| Clay-M | 4.1 | 4.8 | 2.8 | —— | —— |